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1.—RESULTS OF A MICROSCOPIC STUDY OF SOME SOIL COLLOIDS.

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INTRODUCTION.

In two previous publications (1) and (2) J. Shearer and the author have described the X-ray analysis of some soil colloids from Western Australia and Tasmania. In this paper an account is given of a microscopic examination of these soil colloids.

In the first publication cited, the crystalline content of colloid separations (effective diameter 2μ and less) from two soil samples from the Salmon Gums district in Western Australia was determined. These two samples, designated as A 1346 and A 1351, represent the subsoil and deep subsoil respectively.

In the second publication cited, the crystalline content of colloid separations (effective diameter 1.4μ and less) from four soil samples from Tasmania was determined. The details regarding the source of these four samples are :

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|-------------|---|
| 551 and 560 | Colloid separations from soils from Illawarra in northern Tasmania ; |
| 499 | Colloid separation from a basaltic soil of north-west Tasmania ; |
| 222 | Colloid separation from a soil from the Huon valley in southern Tasmania. |

The final X-ray conclusions regarding the composition of these soil colloid fractions are contained in Table 1. The terms " kaolinite," " montmorillonite," and " mica " have been used in this table and in the remainder of this paper, for brevity, to designate a mineral belonging to the kaolinite group, the montmorillonite group and the mica group respectively. This is necessary because at present it is not possible to identify minerals within a group in the diffraction pattern of a soil colloid.

TABLE 1.
COMPOSITION OF THE SOIL COLLOIDS.
(Results of X-ray analysis.)

Mineral.	Tasmanian.				West Australian.
	222.	551.	560.	449.	A 1351 and A 1346.
Quartz ...	Much	←— Much	←— Much	Very Little	Possible trace
Kaolinite. ...	Little	Much	Much	Much	Little
Mica ...	—	—	—	—	Much
Montmorillonite	Little	Possible trace	Possible trace	—	—
Haematite ...	—	Very Little	Very Little	Little	—

Note : An arrow (—>) indicates increasing amounts of quartz. A similar assessment with regard to kaolinite in 551, 560 and 499 could not be made.

The soil colloid fractions from Western Australia were separated according to the International Method which gives particles of an effective diameter of 2μ and less. The soil colloid fractions from Tasmania were separated according to the former British system which gives particles of an effective diameter of 1.4μ and less. From the point of view of this study the main differences in these two methods of preparation are (1) the Tasmanian soil colloid fractions are of a finer grade than the soil colloid fractions from Western Australia, and (2) the former were oven-dried whereas the latter were air-dried. As supplied for examination the local material was still attached to the filter paper and had to be removed by scraping with a glass slide. The Tasmanian material was supplied in a loose powdered form.

For further information regarding modifications introduced into the two methods of separation used, reference may be made to the original articles.

SOIL COLLOID FRACTIONS FROM THE SALMON GUMS DISTRICT IN WESTERN AUSTRALIA.

Both soil colloid fractions A 1351 and A 1346 when viewed under the microscope were seen to be in the form of particles up to 50μ and greater in diameter. Since (it was claimed that) the clay separations were composed of particles of 2μ and less in effective diameter, it was assumed that the particles viewed beneath the microscope were aggregates of the soil colloid particles. In that they exhibited definite optical properties, all these aggregates, in both samples, closely resembled fragments of a single mineral. The optical properties of these aggregates, it is considered, are due to the individual components exhibiting uniformity of crystallographic orientation. This orientation of soil colloid and clay particles into aggregates after drying was originally observed by Hendricks and Fry (3) and has been further investigated by Bray, Grim, and Kerr (4). The aggregates appearing in the soil colloid fractions of A 1351 and A 1346 are larger than those noted by Bray, Grim, and Kerr.

In samples A 1351 and A 1346 the perfection of the uniformity of orientation of the individual components into aggregates is amazing. It was not possible to identify more than one type of aggregate. This indicated that such aggregates were characteristic of the sample as a whole.

The refractive indices were determined on aggregates by immersion in mixtures of clove oil and α -monochloronaphthalene.

The main differences between the two colloid separations A 1346 and A 1351 is one of colour. Under the microscope A 1346 is light brown ; A 1351 is chocolate brown. All the material of both samples is doubly refracting ; A 1351 being the more birefringent. The extinction of the aggregates is in all cases sharp. The indices in different directions were easily determinable. The optical interference figure of aggregates of both samples is biaxial negative and the axial angle is small. These figures are similar in character to those mentioned in the original work of Hendricks and Fry (3). The optical properties of aggregates of the soil colloid fractions A 1351 and A 1346 are summarised in Table 2.

TABLE 2.

OPTICAL DATA RELATING TO THE SOIL COLLOID FRACTIONS FROM WESTERN AUSTRALIA.

Optical Properties.	A 1346 (Water Dispersed).	A 1351 (Water Dispersed).
α	1.562	1.555
γ	1.572	1.570
$\gamma - \alpha$	0.010	0.015
2V	Small	Small
Optical character	(-)	(-)

It may be seen from Table 3 that these optical properties are not those of any common clay mineral. X-ray evidence indicates that both the soil colloid samples are composed of a mixture of two clay minerals, one belonging to the mica group and the other to the kaolinite group. If the aggregates under examination are to be considered representative of the sample as a whole, then these optical properties could result from an aggregation of the above two clay minerals. That two clay minerals *belonging to different groups* may be closely intergrown into aggregates which possess uniformity of crystallographic orientation has already been noted by Bray, Grim, and Kerr (4). Hence the optical properties of aggregates of the soil colloid fractions A 1351 and A 1346 are consistent with the X-ray conclusions.

TABLE 3.

OPTICAL PROPERTIES OF CLAY MINERALS.

KAOLINITE GROUP.

Mineral.	γ	α	$\gamma - \alpha$	2V.	Sign.
Kaolinite	1.570—1.560	1.563—1.553	0.006	24°—50°	(—)
Nacrite	1.566—1.563	1.560—1.557	0.006	40°—90°	(+) or (—)
Dickite	1.556	1.560	0.006	52°—80°	(+)
Halloysite	Mean Index	1.561—1.549	Very slight		
Hydrous Halloysite	Mean Index	1.542—1.526	Very slight.		

MONTMORILLONITE GROUP.

Mineral.	γ		α	$\gamma - \alpha$		2V.	Sign.
Montmorillonite ...	1.530	1.500	1.505—1.470	0.035	0.025	7°—25°	(—)
Beidellite ...	1.560	1.530	1.530—1.500	0.035	0.025	Small	(—)
Nontronite ...	1.610	1.560	1.575—1.530	0.035	0.030	Small	(—)
Saponite ...	Indices of refraction similar to Montmorillonite						

Mica Group.

Mineral.	γ		α	$\gamma - \alpha$		2V.	Sign.
Illite ...	1.605—1.565		1.570—1.535	0.035—0.030		Small	(—)

Note: This Table is based on that published by Grin (5).

SOIL COLLOID FRACTIONS FROM TASMANIA.

In general the remarks made in the previous section as regards aggregation of soil colloid material after drying apply also to the Tasmanian soil colloid fractions. When viewed under the microscope all four samples were seen to be in the form of particles up to 50μ and greater in diameter. In view of what has been previously stated, these particles are considered to be aggregates of the soil colloid particles which have an effective diameter (in this case) of 1.4μ and less. Sample 222 is, however, the only fraction which shows any deducible uniformity of crystallographic orientation of individual components. The three samples, 551, 560, 499, and the greater part of 222, are dark between crossed nicols. In such samples a mean index of refraction only was determined. Variations from the mean value are small.

The soil colloid samples 551, 560, and 499 are very similar. Aggregates formed from each of these three samples are characterised by their colour, high indices of refraction, and apparently isotropic character. The colour of 499 under the microscope is chocolate brown, that of 551 and 560 dark brown. The high indices of refraction are probably due to the high iron content of the soil colloid samples. This iron content does not enter the structure of the clay minerals present in the soil colloids (a conclusion arrived at from the X-ray analysis) but probably coats the colloid particles in the form of iron oxides or hydroxides. Such a possibility has already been noted by Nagelschmidt (6). These indices of refraction, therefore, clearly do not represent the refractive index of the clay mineral shown to be present by X-ray examination. Hendriks and Fry (3) have noted that a high iron content in a soil colloid fraction tends to mask the doubly refracting material present. In view of the X-ray conclusion that the clay mineral present in these three colloid fractions is predominantly kaolinite, it is believed that, in the absence of a high iron content, aggregates formed from each of these three colloid fractions would still possess only a very weak birefringence. Hence, generally speaking, the optical data relating to aggregates of the soil colloid fractions 551, 560, and 499 (which are summarised in Table 3) are consistent with the X-ray conclusions.

Sample 222 is different from the other Tasmanian samples in that the colour beneath the microscope is greyish-yellow, the indices of refraction are lower, and the aggregates are more birefringent. Many aggregates are dark between crossed nicols but some show fairly uniform crystallographic orientation of individual components and for these $\gamma - \alpha = 0.005$. Such aggregates failed to show interference figures sufficiently satisfactory for the determination of the optical character. X-ray evidence indicates that kaolinite and montmorillonite are the clay minerals present in this soil colloid fraction. In view of the fact that the three colloid fractions 551, 560, and 499, containing kaolinite as the only clay mineral are non-birefringent, the slightly birefringent character of 222 (Table 4) is consistent with the above X-ray conclusion. That is, if the aggregates under examination are to be considered representative of the sample as a whole, then these two minerals (kaolinite and montmorillonite) are occasionally closely intergrown into aggregates which possess uniformity of crystallographic orientation.

TABLE 4.

OPTICAL DATA RELATING TO THE SOIL COLLOID FRACTIONS FROM TASMANIA.

Sample.	α	Mean Refractive Index.	γ	$\gamma - \alpha$
222	1.567	...	1.572	0.005
551	...	1.605
560	...	1.618
499	...	1.655

RE-DISPERSION AND RE-AGGREGATION OF THE SOIL COLLOIDS.

In view of criticism of the observations made in this study that firstly the particles examined were aggregates (of a size up to 50μ and greater in diameter) of soil colloid particles rather than original single crystals which had not been broken down in the preparation, and that secondly two clay minerals belonging to different groups may be closely intergrown into aggregates which possess uniformity of crystallographic orientation, it was of interest to restore the particles to their original suspended state where they had an effective diameter of 2μ and to reproduce the aggregates by removing the particles from suspension. This applied particularly to the soil colloid fractions from Western Australia which exhibit a well marked crystallinity.

Re-dispersion.—The re-dispersion of one of these samples (A 1351) was undertaken by Mr. Burvill, of the State Department of Agriculture, who was responsible for the original separation of the West Australian soil colloids. By repeating precisely the procedure adopted in the preparation, Mr. Burvill was able to re-disperse the greater part of the soil colloid sample A 1351. The individual particles of this suspended material were not visible under high power magnification. That some material was not re-dispersed is of little consequence when it is realised that, as a result of the grinding a sample receives in a mechanical analysis, the amount of clay recorded is rather arbitrary.

Re-aggregation.—By removing the clay particles from suspension in the same manner as originally carried out by Mr. Burvill, the author was able to reproduce the state of large-sized crystal aggregates in which the sample originally existed. These aggregates again exhibited a well marked uniformity of crystallographic orientation.

Undoubtedly then the large-sized particles used in the optical examination were not original unbroken particles, but on the contrary, were aggregates of soil colloid particles (the particles being of 2μ and less in effective diameter) which had formed after the dispersion of the material.

OBSERVATIONS ON THE PROCESS OF AGGREGATION OF SOIL COLLOIDS.

A series of observations were made with a view to determining the particular stage at which the soil colloid suspension tended to aggregate. In this study (which was carried out on the re-dispersed material of A 1351) colloid particles in suspension were allowed to settle on a flat surface (under various conditions) and the collected material was kept constantly under examination, as the water content was slowly removed by evaporation. It was noted, firstly, that the particles settled in such a manner that all the material collected remained dark between crossed nicols no matter whether evaporation was slow or fast, whether flocculated or unflocculated material was used. Secondly, the definition of the interference figure improved as the material lost water. The interference figure became only definitely recognizable when the sample first showed the production of contraction cracks. This is in conformity with the observations of other workers (previously noted) that individual particles existing in a soil colloid suspension tend to orient themselves after drying into aggregates which possess uniform optical properties. That the whole surface was set in an optically uniform manner was seen from the fact that the interference figure produced was the same for all the material collected. This is completely in accord with Grim's observations (7) on the production of ordered aggregates after the drying out of clay particles which had been allowed to settle on a slide immersed in a clay suspension.

The interference figure (which was particularly good for that material which had lost the largest amount of water) had the same characteristics as are summarised in Table 2 for both original samples of A 1346 and A 1351. These properties indicate that the aggregates are superimposed basal plates (with $\beta = \gamma$), normal to which is the acute bisectrix (a). In view of the X-ray conclusions that the soil colloid under examination (A 1351) contained two clay minerals, mica and kaolinite, these observations provide additional evidence that two clay minerals belonging to different groups may be closely intergrown into aggregates which possess uniformity of crystallographic orientation.

SUMMARY.

An account is given of the results of a microscopic examination of soil colloid materials from Western Australia and Tasmania. These results are consistent with the mineralogical composition as deduced from X-ray analysis. The methods employed and the observations made are in conformity with those of other workers in this field.

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BIBLIOGRAPHY.

- (1) Shearer, J., and Cole, W. F.—“Analysis of Soil Colloids by X-ray Diffraction Methods.” *Jour. Roy. Soc. W. Aust.*, 1940, XXVI., p. 121.
- (2) Shearer, J., and Cole, W. F.—“X-ray Analysis of some Tasmanian Soil Colloids.” *Jour. Roy. Soc. W. Aust.*, 1940, XXVI., p. 133.
- (3) Hendricks, S. B., and Fry, W. H.—“The Results of X-ray and Microscopic Examination of Soil Colloids.” *Soil Sci.*, 1930, XXIX., pp. 457-479.
- (4) Bray, R. H., Grim, R. E., and Kerr, P. F.—“Application of Clay Mineral Technique to Illinois Clay and Shale.” *Bull. Geol. Soc. Amer.*, 1935, XLVI., pp. 1909-1926.
- (5) Grim, R. E.—“Properties of Clay.” Recent Marine Sediments, A Symposium, London.
- (6) Nagelschmidt, G.—“The Identification of Minerals in Soil Colloids.” *Jour. Agr. Sci.*, 1939, XXIX. [4], pp. 477-501.
- (7) Grim, R. E.—“The Petrographic Study of Clay Minerals. A Laboratory Note.” *Jour. Sed. Petr.*, 1934, IV. [1], pp. 45-46.

NOTE ADDED IN PROOF-READING.

Since this paper was first prepared the author has been successful in obtaining characteristic reflections from oriented aggregates of soil colloid samples. A film of an oriented aggregate of sample A 1351 shows strong basal reflections at 7 Å and 10 Å indicating the presence of both kaolinite and mica in the same aggregate and thus fully confirming the conclusions reached above on the aggregation of soil colloids.
